

Title: : **BIAXIALLY ORIENTED POLYPROPYLENE FILM,
METALLIZED BIAXIALLY ORIENTED
POLYPROPYLENE FILM AND CONDENSER**

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ABSTRACT

PROBLEM TO BE SOLVED: To enhance heat resistance and vapor deposition processability by specifying the isotacticity and isotactic pentad ratio of a biaxially oriented polypropylene film and also specifying the wet tension of at least one surface of the film.

SOLUTION: The isotacticity of a biaxially oriented polypropylene film is 98-99.5%. Herein, the isotacticity is defined by the ratio of the wt. of an insoluble component to the wt. of the film before extraction when the film is extracted with boiling n-heptane. The stereoregularity of the biaxially oriented polypropylene film is evaluated by the pentad ratio due to the absorption peak of a methyl group measured by ¹³C-NMR and the isotactic pentad ratio of the biaxially oriented polypropylene film exceeds 99%. In this biaxially oriented polypropylene film, the wet tension of at least one surface thereof is 35-50mN/m.

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Epitome

(57) [Abstract]

[Technical problem] The small metalization biaxial orientation polypropylene film of a surface-electrical-resistance value and the capacitor which raised the long-term thermal resistance in an elevated temperature are offered by excelling in the biaxial orientation polypropylene film excellent in thermal resistance and vacuum evaporation workability, and self heel nature.

[Means for Solution] An isotactic pentad molar fraction exceeds [AISO tacticity] 99% 98 to 99.5%. The biaxial orientation polypropylene film and AISO tacticity whose wetting tension of one [at least] field is 35 – 50 mN/m 98 – 99.5%, The metalization film with which the metal layer whose relation between surface electrical resistance R and optical density OD is $R-OD < 15$ was formed at least in one side of the biaxial orientation polypropylene film with which the isotactic pentad molar fraction exceeded 99%, and the capacitor using it.

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CLAIMS

[Claim(s)]

[Claim 1] The biaxial orientation polypropylene film which it is the polypropylene film by which biaxial orientation was carried out, the AISO tacticity of this polypropylene film is 98 – 99.5%, and an isotactic pentad molar fraction exceeds 99%, and is characterized by the wetting tension of one [at least] field being 35 – 50 mN/m.

[Claim 2] The metalization biaxial orientation polypropylene film which a metal layer is formed in one [at least] field of the biaxial orientation polypropylene film with which AISO tacticity is 98 – 99.5%, and the isotactic pentad molar fraction exceeded 99%, and is characterized by the surface electrical resistance R of this metal layer (ω /**) and the relation of optical density OD being $R-OD < 15$.

[Claim 3] The capacitor characterized by winding or carrying out a laminating and becoming about a metalization biaxial orientation polypropylene film according to claim 2.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the capacitor which was excellent in a biaxial orientation polypropylene film and self heel nature excellent in especially vacuum evaporation workability about the biaxial orientation polypropylene film excellent in thermal resistance, the metalization biaxial orientation polypropylene film with specific surface electrical resistance and the relation of optical density, and the capacitor using it, and was excellent in the thermal resistance using the small metalization biaxial orientation polypropylene film and small it of a surface-electrical-resistance value as a dielectric, and a dielectric-breakdown-proof property.

[0002]

[Description of the Prior Art] A biaxial orientation polypropylene film is excellent in optical properties, such as transparency and glossiness, and is further used for the package application, the capacitor application, etc. extensively by steam barrier property ability, the outstanding electrical property, etc.

[0003] Although this biaxial orientation polypropylene film was one of the typical materials used as a dielectric of a film capacitor, since thermal resistance was low as compared with the polyester film which is another typical material, the maximum service temperature as a capacitor was restricted to about 85 degrees C. The reason is that there was a case where the dielectric-breakdown reinforcement which should be the features of a polypropylene film essentially fell rapidly, and it became impossible to be especially equal to prolonged use from the amorphism section of a film, the effect of a foreign matter, etc. when service temperature became an elevated temperature.

[0004] On the other hand, with the miniaturization of an electric apparatus, high-density-izing and elevated-temperature-izing of a component progress, and demand that the maximum service temperature of the conventional polypropylene film capacitor wants to rise further is becoming strong. For that, moreover, the engine performance needed to be maintained at the elevated temperature at the long period of time rather than 85 degrees C which is the maximum service temperature of the conventional polypropylene film capacitor.

[0005] Moreover, as for a polypropylene film capacitor, the metal layer as an electrode which generally becomes one field of a film from aluminum, zinc, or these alloys by vacuum evaporation is formed (henceforth metalization), and winding **** carrying out the laminating of this metalization film, and making it into a capacitor element is performed.

[0006] Therefore, in order to obtain the capacitor excellent in the thermal resistance which passed through the above-mentioned process, excelling in workability-proof, such as the slipping nature of that the metal layer by that the film electrical property in an elevated temperature is excellent and (1) (2) vacuum evaporation is formed in homogeneity, excel in self heel nature, and a surface-electrical-resistance value is small, (3) vacuum-evaporation processing, and

capacitor element creation time, was called for.

[0007] The giant-molecule insulating material which ash content was low to JP,6-236709,A, was excellent in workability since ebullition n-heptane extractives were 1 - 10 % of the weight, and was excellent in the electric insulation from a room temperature to 80 degrees C is indicated to such a technical problem, and there is suggestion that 90% or more of thing has the desirable isotactic pentad molar fraction of the ebullition n-heptane insoluble section.

[0008] Moreover, as well as JP,7-25946,A, ebullition heptane insoluble matter is 96 % of the weight or more especially preferably 80% of the weight or more, and there is an indication of the propylene polymer which has the isotactic pentad molar fraction of this ebullition heptane insoluble element in the range of 0.970-0.995, and the Plastic solid using this.

[0009] However, as indicated by these, the biaxial orientation polypropylene film with the only high isotactic pentad molar fraction of ebullition n-heptane insoluble matter of the dielectric-breakdown-proof property in the elevated temperature exceeding 85 degrees C which this invention aims at, and the long-term thermal resistance of the capacitor element using this film as a dielectric was inadequate. That is, although the high biaxial orientation polypropylene film of the stereoregularity by the above-mentioned Prior art had the high isotactic pentad molar fraction of the ebullition n-heptane insoluble section so so, since the isotactic pentad molar fraction of n-heptane extractives was low, its isotactic pentad molar fraction as a film was low as a result, and stereoregularity was inadequate [the film]. Moreover, the so-called biaxial orientation polypropylene film of high crystallinity with very high AISO tacticity has come to be established as a useful technique industrially [film production nature is very bad and] for manufacturing the biaxial orientation polypropylene film excellent in thermal resistance and a dielectric-breakdown-proof property, although stereoregularity is inadequate therefore.

[0010] In order to cancel these faults, the crystalline polypropylene film which was excellent in the moldability that the whole quantity of the extract-ed which there is an isotactic pentad molar fraction in JP,4-28727,B in the range of 0.960-0.990, and carried out sequential sampling by the ebullition n-hexane and the ebullition n-heptane considers as 3.0 - 6.0% is proposed. However, this polypropylene film did not have an enough isotactic pentad molar fraction, and its dielectric-breakdown-proof property in an elevated temperature was inadequate.

[0011] Furthermore, it has specific heat deflection temperature and Young's modulus, and to JP,5-217799,A, degree of crystallinity is high, and the vacuum evaporatio film capacitor using the high rigidity vacuum evaporatio metalization film which vapor-deposited the metal on the good high rigidity polypropylene film of stereoregularity is proposed. However, stereoregularity here was at most about 90%, and was inadequate. [of the dielectric-breakdown property in an elevated temperature]

[0012] Furthermore, the metalization polypropylene film whose rate of a heat shrink in 120 degrees C is 0.8% or less in 4.0% or less and the cross direction in the die-length direction is indicated by JP,7-50224,A. However, it was not able to be said that the AISO tacticity and the stereoregularity of this film not necessarily had an enough dielectric-breakdown-proof property in the elevated temperature which is the thing of the conventional level and is the purpose of this invention for corresponding to a future advanced demand.

[0013]

[Problem(s) to be Solved by the Invention] this invention person etc. controlled the AISO tacticity and the stereoregularity of a polypropylene film to altitude, rationalized surface wetting tension, found out that the capacitor excellent in the thermal resistance which could not be attained with the conventional technique by controlling the surface electrical resistance and optical density of a metalization layer, and a dielectric-breakdown-proof property could be obtained, and resulted in this invention.

[0014] The purpose of this invention is to offer the biaxial orientation polypropylene film which was excellent in thermal resistance and was excellent in vacuum evaporatio workability.

[0015] Other purposes of this invention are to offer the capacitor which was excellent in self heel nature and was excellent in the thermal resistance using the small metalization biaxial orientation polypropylene film and small it of a surface-electrical-resistance value, and a dielectric-breakdown-proof property.

[0016]

[Means for Solving the Problem] The polypropylene film of this invention which attains the above-mentioned purpose is a polypropylene film by which biaxial orientation was carried out, and the AISO tacticity of the polypropylene film is 98 – 99.5%, an isotactic pentad molar fraction exceeds 99%, and it is characterized by the wetting tension of one [at least] field being 35 – 50 mN/m.

[0017] Moreover, the metalization biaxial orientation polypropylene film of this invention A metal layer is formed in one [at least] field of the biaxial orientation polypropylene film with which AISO tacticity is 98 – 99.5%, and the isotactic pentad molar fraction exceeded 99%. It is that to which the surface electrical resistance R of the metal layer (ω/ω) and relation of optical density OD are characterized by being $R-OD < 15$. Or AISO tacticity is 98 – 99.5%, and an isotactic pentad molar fraction exceeds 99%. It is characterized by coming to form the metal layer whose relation between surface electrical resistance R (ω/ω) and optical density OD is $R-OD < 15$ in one [at least] field of a biaxial orientation polypropylene film whose wetting tension of one [at least] field is 35 – 50 mN/m.

[0018] Furthermore, about this metalization biaxial orientation polypropylene film, a laminating is wound or carried out and the capacitor of this invention becomes.

[0019]

[Embodiment of the Invention] Although the biaxial orientation polypropylene film of this invention is a biaxial oriented film which mainly consists of a homopolymer of polypropylene, in the range which does not check the purpose of this invention, the copolymerization component by other unsaturated hydrocarbon etc. may be contained, and you may act as Brent of other polymers. As a copolymerization component, ethylene, 1-butene, 1-pentene, 3-methyl pentene – 1, the 3-methylbutene – 1, 1-hexene, 4-methyl pentene – 1, 5-ethyl hexene – 1, 1-octene, 1-decene, 1-dodecen, a vinyl cyclohexene, styrene, allyl compound benzene, cyclopentene, norbornene, 5-methyl-2-norbornene, etc. are mentioned, for example. As for less than [1mol%] and a blend object, less than [1wt%] is [the amount of copolymerization] desirable from vacuum evaporatio workability, a dielectric-breakdown-proof property, and a heat-resistant point.

[0020] In this invention, the AISO tacticity of a biaxial orientation polypropylene film is 99.5% or less especially in respect of the crosswise ductility at the time of biaxial stretching. The weight of the insoluble matter to the film weight before an extract at the time of extracting by the ebullition n-heptane looks a film like [AISO tacticity] comparatively, and it is defined more as it here. In case a biaxially oriented film will be manufactured as it is in JP,6-236709,A if AISO tacticity is too high, ductility is bad and film production becomes it is remarkable and difficult. Moreover, especially AISO tacticity is 98% or more in respect of vacuum evaporatio workability and the heat-resistant defeat nature at the time of vacuum evaporatio when film thickness is set to less than 5 micrometers in more detail. Thus, if AISO tacticity is 98% or more and 99.5% or less, also in the case of a very thin film, good extension can be performed like less than 5 micrometers, and a high level can be maintained in the heat-resistant defeat nature at the time of vacuum evaporatio, film thickness enables the miniaturization using a thin film, and the highly efficient capacitor which is not in the former can be realized. If AISO tacticity is too small, it may be inferior to thermal resistance, and Siwa by the heat defeat at the time of vacuum evaporatio may occur. Desirable AISO tacticity is 98.7 – 99.5% because of good film production nature and vacuum evaporatio workability, and a dielectric-breakdown-proof property, and further 98.7 – 99.3% is desirable.

[0021] In order to consider as the biaxial orientation polypropylene film which has such AISO tacticity, the so-called rate of the low molecular weight constituent which is easy to melt into the ebullition n-heptane of the polypropylene resin which is a raw material, and an atactic part with low stereoregularity can adopt the approach of choosing ** for a low thing moderately.

[0022] In this invention, the pentad molar fraction by the absorption peak of the methyl group measured by ^{13}C -NMR can estimate the stereoregularity of a biaxial orientation polypropylene film. Generally, there are mmmm, mmmr, rmmr, ..., rrrr, mrrr, mrrm, etc. in the conformation of five repeat units (pentad) in a polypropylene chain. Here, m shows meso (meso) and r shows the

conformation of racemo (rasemo).

[0023] The pentad molar fraction of a biaxial orientation polypropylene film can ask for the ratio of the segment which has each above-mentioned conformation from ^{13}C -NMR, as it is in T.Hayashi's and others report (Polymer, and [29,138-143] (1988)) etc. the conformation of mmmm [as opposed to / among these / the absorption intensity of all methyl groups] -- comparatively -- that is, an isotactic pentad molar fraction (it may abbreviate to Following mmmm) is defined as the sum of three HEPUTADDO molar fractions, $m(\text{mmmm})$ m, $m(\text{mmmm})$ r, and $r(\text{mmmm})$ r. The isotactic pentad molar fraction mmmm of the biaxial orientation polypropylene film of this invention exceeds 99%. Since such a film consists of polypropylene which consisted of molecules with a very long isotactic segment, the film of high crystallinity and high thermal resistance excellent in vacuum evaporatio no possibility, and a dielectric-breakdown-proof [quantity] property can be given.

[0024] mmmm of the biaxial orientation polypropylene film of this invention is 99.1% or more preferably in respect of vacuum evaporatio no workability, high thermal resistance, and a dielectric-breakdown-proof [quantity] property, is 99.2% or more more preferably, and is 99.3% or more still more preferably.

[0025] In order to give such stereoregularity, it is effective to control to altitude the stereoregularity of the polypropylene resin which is a raw material. It is attained as an approach of creating such a raw material by the catalyst systems (the solid-state catalyst, external addition electron-donative compound) and such purity at the time of carrying out the polymerization of the polypropylene. Although the inclination for mmmm of a biaxial orientation polypropylene film to become high in the thing which has high mmmm of the polypropylene resin of a raw material is accepted, in order that the heat deterioration of the degree of pole within the extrusion system of a raw material may also reduce mmmm, the structural device of avoiding long duration stagnation of the raw material in a hot extrusion system and extrusion conditions are chosen suitably.

[0026] Moreover, in the biaxial orientation polypropylene film of this invention, the wetting tension of the front face of one [at least] of these is 35 - 50 mN/m. When wetting tension was too small, and the metal layer used as an electrode is hard to be formed in homogeneity, the electric resistance of an electrode section becomes large and it is processed into a capacitor element, dielectric loss becomes large, degradation is further accelerated by generation of heat, and the life as a capacitor element may become short. On the other hand, if wetting tension is too large, the slipping nature of a film will worsen, and it becomes defects, like Siwa enters at the time of vacuum evaporatio no processing, and produces component creation time un-arranging, and the property of a component may be worsened. The still more desirable range of wetting tension is 37 - 48 mN/m, and is 38 - 45 mN/m most preferably.

[0027] in order to make surface wetting tension into such range in this invention -- corona discharge treatment and plasma treatment -- desirable -- applicable -- the corona discharge treatment in the inside of an atmospheric-air ambient atmosphere, nitrogen-gas-atmosphere mind, a carbon-dioxide-gas ambient atmosphere, and these mixed ambient atmospheres -- from a viewpoint of productivity -- suitable -- a film production process and the last process of vacuum evaporatio no -- you may carry out by any. Although processing reinforcement is adopted in the range of 5 - 50 W-min/m², since a treatment effect changes with processing ambient atmospheres, in order to consider as the wetting tension of this invention, it is required to adjust processing reinforcement suitably.

[0028] In the embodiment of this invention, forming a metal layer in a biaxial orientation polypropylene film, and processing it into a metalization biaxial orientation polypropylene film is adopted preferably because of the miniaturization of a capacitor element. In this case, although especially the metal that constitutes a metal layer is not limited, it is desirable independent or to use aluminum, zinc, copper, tin, silver, nickel, etc. by concomitant use in respect of the endurance of a metalization layer, and productivity, and aluminum, zinc, etc. are preferably used especially from the point of economical efficiency.

[0029] The approach of forming a metal layer in the biaxial orientation polypropylene film of this invention is not especially limited, although vacuum evaporation technique, the sputtering

method, the ion beam method, etc. are mentioned.

[0030] In this invention, the desirable surface-electrical-resistance value R of a metalization biaxial orientation polypropylene film is the range of 1–20ohms / **, and are 1.2–15ohm/** more preferably. When a membrane resistance value is too small, the thickness of the vacuum evaporation film is thick, at the time of vacuum evaporation, heat defeat may arise and hole Aki etc. may occur with a surface crater-like fault or the thin film around 4 micrometers. Moreover, if a membrane resistance value is too large, a surface-electrical-resistance value will become large, the dielectric loss as a capacitor element becomes large, and it is easy to lead to destruction of a component. In order to make a membrane resistance value into this range, the approach of controlling by the monitor of the membrane resistance value at the time of vacuum evaporation is adopted preferably.

[0031] As for the optical density OD of the biaxial orientation polypropylene film of this invention, being chosen in 0.5–3 is desirable. The range of optical density is 0.8–2 still more preferably. This invention is defined as optical density by the logarithm of the inverse number of the light transmission in the specific wavelength of a visible region. When optical density is too large, the problem of the heat defeat at the time of vacuum evaporation may arise. If optical density is too small, the problem to which a surface-electrical-resistance value becomes large may arise.

[0032] In this invention, it is important that the surface-electrical-resistance value R and optical density OD have the relation of $R-OD < 15$ (omega/**). Although OD becomes large in proportion to the thickness of a vacuum evaporation metal and R generally becomes small according to it, $R-OD$ is too large, i.e., considering R of a vacuum evaporation metal, when OD, i.e., thickness, is large, the self heel nature of a metalization biaxial orientation polypropylene film worsens, and a problem arises in the property as a capacitor element.

[0033] As a cause by which $R-OD$ becomes large, since the adhesive property of a vacuum evaporation metal and an original fabric biaxial orientation polypropylene film front face is bad, it is possible that film formation of the vacuum evaporation metal is not carried out at homogeneity. For this reason, when it is going to acquire a certain specific membrane resistance value, the problem actuation in which thickness evaporates the vacuum evaporation metal of the that defective partial circumference by discharge breakdown at a self heel, i.e., capacitor element creation time, in the very small electrical insulation defect of an original fabric film, and makes it inactive by becoming large becomes impossible arises. Moreover, in order to raise self heel nature, when it is going to make thickness small, since the homogeneous membrane of a vacuum evaporation metal is not formed, a membrane resistance value may rise rapidly, dielectric loss may become large as a capacitor element, and use may not be borne. The more desirable range of $R-OD$ is below 13ohms / **. In order to make $R-OD$ into such range, it is important to control the wetting tension on the front face of an original fabric proper and to keep good the degree of vacuum at the time of metal vacuum evaporation.

[0034] In this invention, other than a type, the specification of a margin (part without the metal layer prepared in the field which forms a metal layer for the electric insulation purpose etc.) established when forming a metal layer in a biaxial orientation polypropylene film can be adopted according to the purposes, such as various things which established the fuse device, and is not usually limited especially.

[0035] In the above-mentioned metalization biaxial orientation polypropylene film, a laminating is wound or carried out and the capacitor of this invention is created.

[0036] The format of the capacitor of this invention is not especially limited, although dry type, an oil immersion ceremony, etc. are held.

[0037] As for the sum of the rate of a heat shrink of the direction of a machine at the time of heating during 120-degree-C 5 minutes of the biaxial orientation polypropylene film of this invention, and a metalization biaxial orientation polypropylene film, and the cross direction, it is desirable that it is 1 – 4% of range. If the rate of a heat shrink is too large, at the time of the metal stratification as an electrode, Siwa goes into a lifting film roll, or since the mechanical deformation by the heat of capacitor element creation time is too large, stress generates a dimensional change in the inside of a film, and the contact section with/or an external electrode,

and the capacity fall of a capacitor may become large or may result at destruction of a component. Conversely, when the rate of a heat shrink is too low, the volume tightness according to heat treatment of capacitor element creation time becomes inadequate, and it has a bad influence on gestalt holdout or capacity rate of change. The above-mentioned sum is 1.5 – 3.5%, and the desirable rate of a heat shrink has further 1.8 – 3%, and further 2 – 2.8% of desirable range. In order to make the rate of a heat shrink into such range, the conditions at the time of film production are very important.

[0038] It is suitable for the biaxial orientation polypropylene film which has conventional AISO tacticity and conventional mmmm that the cast of the biaxial orientation polypropylene film of this invention is carried out to 40–85 degrees C at lower casting drum temperature to the cast having been carried out at the casting drum temperature of 85 degrees C or more as indicated by JP,7-50224,A. If casting drum temperature is too high, crystallization of a film will advance too much, and the rate of a heat shrink becomes [whether extension at a next process becomes difficult and] too much. Moreover, as for the conventional biaxial orientation polypropylene film, it was common that the direction extension temperature of a machine of 140 degrees C or less and the crosswise extension temperature of 160 degrees C or less were adopted, and since orientation fell at the extension temperature exceeding these temperature, it was difficult to maintain the elastic modulus as a biaxial orientation polypropylene film. With the biaxial orientation polypropylene film of this invention, the direction extension temperature of a machine of 140–150 degrees C and the crosswise extension temperature of 160–165 degrees C are preferably adopted, in order to obtain the rate of a heat shrink made into the purpose, with the elastic modulus as a biaxial orientation polypropylene film maintained. If these extension temperature is too low, the rate of a heat shrink will become large too much. As for heat treatment temperature while making the cross direction ease furthermore, it is also effective to consider as 150–160 degrees C. If heat treatment temperature is too low, the rate of a heat shrink will become large too much, and if too high, the rate of a heat shrink will become small too much.

[0039] In this invention, the thickness of a biaxial orientation polypropylene film and a metalization polypropylene film has the desirable range of 2–30 micrometers from the point of film production nature, a mechanical characteristic, and an electrical property, and 2.5–20 micrometers is 2.5–10 micrometers still more preferably more preferably. If the thickness of a film is too small, it may be inferior to dielectric-breakdown reinforcement or a mechanical strength, and damage on the film by heat defeat may occur in *****. When the thickness of a film was too large, and it becomes difficult to produce the film of uniform thickness and it uses as a dielectric for capacitors, since the capacity per volume becomes small, it is not desirable.

[0040] In this invention, although especially the limiting viscosity of polypropylene excellent in the stereoregularity used for a biaxial orientation polypropylene film is not limited, the thing of the range of the point of film production nature to 1 – 10 dl/g is desirable. Moreover, the melt flow rate in 230 degrees C and 2.16kg load has a desirable thing for 2–5g from the point of film production nature / 10 minutes. In order to make limiting viscosity and a melt flow rate into the above-mentioned value, the approach of controlling average molecular weight and molecular weight distribution etc. is adopted.

[0041] In the polymerization process of polypropylene, although it is common to remove this residue after a polymerization if needed, using the compound containing a metal as a catalyst, calculating the amount of the remaining metallic oxide which burned resin completely can estimate this residue, and it calls this ash content. It is desirable that it is 30 ppm or less, it is 25 ppm or less more preferably, and the ash content of the biaxial orientation polypropylene film of this invention is 20 ppm or less still more preferably. If there is too much ash content, the dielectric-breakdown-proof property of a film may fall and the dielectric-breakdown reinforcement of the capacitor using this may fall. Although it is important to use a raw material with few catalyst residues in order to make ash content into this range, the approach of also reducing the contamination from the extrusion system at the time of film production as much as possible, for example, approaches, such as spending bleeding time amount for 1 hour or more, is employable.

[0042] Although the surface roughness of the biaxial orientation polypropylene film of this invention and a metalization biaxial orientation polypropylene film is suitably chosen according to the purpose, the range of 0.02–0.2 micrometers is desirable at the center line average of roughness height. If center line average granularity is too large, when the laminating of the film is carried out, air may enter between layers and it may lead to degradation of a capacitor element. Conversely, if too small, slipping of a film may worsen, and it may be inferior to handling nature.

[0043] The biaxial orientation polypropylene film of this invention and a metalization biaxial orientation polypropylene film may be made to contain a well-known additive, for example, a crystalline-germ agent, an anti-oxidant, a thermostabilizer, a skid agent, an antistatic agent, an antiblocking agent, a bulking agent, a viscosity controlling agent, a coloring inhibitor, etc.

[0044] In these, the class of antioxidant and selection of an addition are important for thermal resistance over a long period of time. The antioxidant added by the biaxial orientation polypropylene film of this invention and the metalization biaxial orientation polypropylene film is the thing of the phenol nature which has steric hindrance nature, among those its at least one sort is desirable because of the scattering prevention at the time of melting push of a with a molecular weight of 500 or more amount of macromolecules type thing. Although various things are mentioned as this example With for example, 2,6-di-*t*-butyl-*p*-cresol (BHT: molecular weight 220.4) 1, 3, 5-trimethyl -2, 4, 6-tris Benzene (3, 5-G *t*-butyl-4-hydroxybenzyl) (For example, Ciba-Geigy Irganox1330: Molecular weight 775.2) Or tetrakis [methylene -3 (3, 5-G *t*-butyl-4-hydroxyphenyl) propionate] methane (for example, Ciba-Geigy Irganox1010: molecular weight 1177.7) etc. Using together is desirable. The total content of these antioxidants has 0.03 – 1% of the weight (300–10000 ppm) of the desirable range to the polypropylene whole quantity. If there are few total contents, it may be inferior to thermal resistance over a long period of time, and if many [too], it may have a bad influence on a capacitor element by blocking under the elevated temperature by the bleed out of these anti-oxidants. A more desirable content is 0.1 – 0.9 % of the weight, and is 0.2 – 0.8 % of the weight still more preferably.

[0045] The situation of partial discharge breakdown at the time of impressing direct current voltage can estimate the self heel nature of the metalization biaxial orientation polypropylene film of this invention. The evaporation field of the vacuum evaporatio metal membrane after partial discharge breakdown is small, and when a vacuum evaporatio metal does not remain around discharge marks, self heel nature is good. That by which signs that the metal remained around the discharge marks by discharge, and the film dissolved are observed may result in dielectric breakdown of the component itself, when self heel nature is bad and processes it into a capacitor. At the time of electric charging at 105 degrees C, dielectric breakdown may be accelerated especially.

[0046] 105 degrees C can estimate the property of the capacitor of this invention with the dielectric loss magnitude after 1.2 to 1.3 times as much alternating-voltage electric charging as the rated voltage of 100 hours, i.e., tandelta. If tandelta is too large, generation of heat of a component is accelerated and it may result in destruction for a short time. Although the manufacture approach of the capacitor which consists of the biaxial orientation polypropylene film of this invention, a metalization biaxial orientation polypropylene film, and it is explained below, this invention is not necessarily limited to this.

[0047] first, the temperature of 220–320 degrees C after having supplied the polypropylene raw material to the extruder, carrying out heating fusion and making a barrier filter let it pass -- the shape of a slit -- from a mouthpiece, it twists around the casting drum maintained at melting extrusion and the temperature of 40–85 degrees C, cooling solidification is carried out, and an unstretched film is made.

[0048] Next, biaxial stretching of this unstretched film is carried out, and it carries out biaxial orientation. As the extension approach, the biaxial-stretching approach is desirable serially. Serially, the extension approach lets pass and carries out the preheating of the unstretched film to the roll kept at 120–150 degrees C first, is extended to through between the rolls which maintained the sheet at the temperature of 140 degrees C – 150 degrees C succeedingly, and established the peripheral-speed difference, extends it 2 to 6 times to a longitudinal direction, and is immediately cooled to a room temperature. Here, it is important for the biaxial orientation

polypropylene film with which mmmm of this invention exceeds 99% to adopt the extension temperature which a heating value is insufficient at the preheat temperature of 130 degrees C or less and the extension temperature of 140 degrees C or less, and starts extension nonuniformity, or is torn, may be unable to produce a film, and exceeds 140 degrees C. Leading the oriented film to a tenter succeedingly, extending 5 to 10 times crosswise at the temperature of 160–165 degrees C, and, giving 2 – 20% of relaxation subsequently to the cross direction, at the temperature of 150–160 degrees C, heat setting is carried out and it rolls round. In this invention, if the temperature of this heat setting is important and is too low, the rate of a heat shrink may become large and it may exceed the range of this invention. [of heat setting temperature]

[0049] Then, if to the field which vapor-deposits, in order to close the adhesive property of a vacuum evaporatio metal to it, corona discharge treatment is performed in air, nitrogen, carbon dioxide gas, or these mixture of gas, and it rolls round by the winder.

[0050] Next, the obtained film is set in a vacuum evaporation system, and the metal according to the purpose is vapor-deposited to predetermined membrane resistance. The slit of this vacuum evaporatio film is carried out, and it considers as the vacuum evaporatio reel of 2 reel pair for making a capacitor element. Then, it winds in the shape of a component, and a heat press is carried out, and it fabricates in the shape of flat, and considers as a capacitor through the metallizing of an edge, lead ejection, and sheathing.

[0051] In addition to the above-mentioned capacitor application, since deformation by the heat at the time of processing of vacuum evaporatio, printing, a lamination, heat sealing, etc. can be suppressed taking advantage of the low rate of a heat shrink, the biaxial orientation polypropylene film of this invention can be used for this as various package applications, laminating it in it with a heat-sealing layer, and can be suitably used also as adhesive tape, a glazing film (print lamination), etc.

[0052] The evaluation approach is as follows at the measuring method of the characteristic value in this invention, and a list.

[0053] (1) AISO tacticity (isotactic index: II)

A sample is extracted by n-heptane with a temperature of 60 degrees C or less for 2 hours, and the additive to polypropylene is removed. A vacuum drying is carried out at 130 degrees C after that for 2 hours. From now on, the sample of weight W (mg) will be taken, and it puts into a Soxhlet extractor, and extracts by the ebullition n-heptane for 12 hours. Next, after it took out this sample and the acetone washed enough, it cooled at 130 degrees C for 6 hours to the back ordinary temperature of vacuum-drying *Perilla frutescens* (L.) Britton var. *crispa* (Thunb.) Decne., weight W' (mg) was measured, and it asked by the degree type.

$II = (W'/W) \times 100 (\%)$

(2) The isotactic pentad molar fraction sample was dissolved in o-dichlorobenzene, and ¹³C-NMR was measured with the resonance frequency of 67.93MHz using JNM-GX270 made from JEOL equipment. Based on the approach (Polymer, and [29,138–143] (1988)) which T.Hayashi and others performed about attribution of the obtained spectrum and count of a pentad molar fraction, each peak was belonged about the spectrum of the methyl group origin, having used the mmmmm peak as 21.855 ppm, and the ratio to a methyl group origin all peak area was expressed as the percentage in quest of the peak area. The detailed Measuring condition is as follows.

[0054] Measurement solvent : o-dichlorobenzene (90wt%) / benzene-D6 (10wt%) measurement temperature : 120–130 degree-C resonance frequency: 67.93MHz pulse width : 10microsec (45-degree pulse)

Pulse repetition time: 7.091sec data point : Count of 32K addition : 8168 measurement mode: It measured using the noise decoupling (3) film thickness dial gage type thickness meter (JIS-B - 7503).

[0055] (4) Wetting tension JIS It measured according to K-6788.

[0056] (5) Viewing estimated the film plasticity of the vacuum evaporatio film after vacuum evaporatio processing of a metal membrane plasticity biaxial orientation polypropylene film, and the following judgments were performed.

[0057] O : there is no unevenness in a metal membrane and the metal membrane is formed in homogeneity.

[0058] x: Unevenness is in a metal membrane.

[0059] (6) Viewing estimated the condition of the roll of the volume riser after vacuum evaporation processing of a vacuum evaporation workability biaxial orientation polypropylene film, and the following judgments were performed.

[0060] O : Siwa has not occurred on a roll.

[0061] x: Siwa has occurred on the roll.

[0062] (7) The transmission density when setting a filter to Visual was measured using the optical-density OD optical-density meter (Macbeth TR927), and the value which deducted the optical density of an original fabric film from the optical density of a metalization film was made into the optical density of a metal layer.

[0063] (8) It measured using 4 terminal probe using the surface-electrical-resistance R surface-electrical-resistance meter (Mitsubishi Petrochemical Loresta-FP).

[0064] (9) The field which is not metalizing the self heel nature metalization biaxial orientation polypropylene film was contacted to the flat electrode plate made from brass, one twice the direct current voltage of rated voltage (in the case of a polypropylene film, it is 45-50v/micrometer) was impressed between the metalization side and the electrode plate made from brass, and partial dielectric breakdown was generated. The dielectric-breakdown part was observed and the following self heel nature was judged.

[0065] O : a metal is not accepted for the diameter of **** formed in the film of dielectric breakdown around a hole by 1mm or less.

[0066] x: The marks of the dissolution of the diameter of a hole of a film by 3mm or more are accepted.

[0067] **: The capacitor element held at the 105 degrees C of the above-mentioned middle (10) tandelta dielectric loss was connected to the alternating current high-pressure regulated power supply made from Kasuga Electrical and electric equipment, the electrical potential difference of 60v/micrometer was impressed, and tandelta of 100 hours after was measured by electrical-potential-difference 250V by the Schering bridge method.

[0068]

[Example] This invention is explained below at a detail based on an example and the example of a comparison.

[0069] (Example 1) The isotactic index II supplies to an extruder that to which the isotactic pentad molar fraction mmmm added 0.3% [of 2,6-di-t-butyl-p-cresol] (BHT), and TERORAKISU [methylene -3 (3, 5-G t-butyl-4-hydroxyphenyl) propionate] methane (Irganox1010) 0.5% 99.5% in the polypropylene raw material whose ash content is 19 ppm 98.8%. It fused at the temperature of 280 degrees C, and extrusion molding was carried out to the shape of a sheet from the mouthpiece T molds, it twisted around the casting drum with a temperature of 70 degrees C, and cooling solidification was carried out. Subsequently, the preheating of this sheet was carried out at 135 degrees C, and it extended to through between the rolls which maintained at the temperature of 143 degrees C succeedingly, and established the peripheral-speed difference, and extended 5 times to the longitudinal direction. Having led the film to the tenter succeedingly, having extended 10 times crosswise at the temperature of 162 degrees C, and, giving 8% of relaxation subsequently to the cross direction, it heat-treated at 158 degrees C, and the biaxial orientation polypropylene film with a thickness of 4 micrometers was obtained. Corona discharge treatment was performed to the pan in atmospheric air by the processing reinforcement of 30 W-min/m², and it considered as wetting tension 41 mN/m. The ash content and the pentad molar fraction of a film which were obtained did not have those values and differences of a raw material. This film was set to the vacuum deposition machine, and it vapor-deposited so that membrane resistance might become 6ohms / ** about aluminum in a corona treatment side. The optical density of this film was 1.8. The slit of this film was carried out and the metalization film with a full [of 38mm] and a margin width of 1mm was obtained. The component volume was carried out using film pair 2 obtained reel, metallizing was carried out to the end face of a component, lead wire was taken out from here, and the capacitor element with a capacity of 5

micro F was created.

[0070] (Example 2) It considered as the wetting tension of 38 mN/m by making into 10 W-min/m² processing reinforcement of the corona discharge treatment of the biaxial orientation polypropylene film created like the example 1. The vacuum plating of aluminium of 6ohms / ** was performed as well as the example 1, and optical density obtained the metalization film of 2.0. The capacitor element was created like the example 1 using this.

[0071] (Example 3) Using the biaxial orientation polypropylene film of the wetting tension of 41 mN/m of an example 1, aluminum was vapor-deposited and the metalization film of optical density 1.2 was obtained. Moreover, surface electrical resistance was 11ohm/**. The capacitor element was created like the example 1 using this.

[0072]

[Table 1]

表 1

	II (%)	mmmm (%)	ぬれ張力 (mN/m)	蒸着加工性	膜形成性	表面抵抗 (Ω/□)	光学濃度	R・OD (Ω/□)	成形性	tandδ
実施例 1	98.8	99.5	41	○	○	6	1.8	10.8	○	0.02
実施例 2	"	"	38	○	○	6	2.0	12	○	0.02
実施例 3	"	"	41	○	○	11	1.2	13.2	○	0.04
比較例 1	98.3	98	41	×	○	6	1.9	11.4	○	破壊
比較例 2	99.8	99.5	32	○	×	6	2.6	15.6	×	破壊
比較例 3	"	"	"	○	×	22	0.9	19.8	○	0.15
比較例 4	"	"	52	×	—	—	—	—	—	—

* 熱食けによるシワの発生

** 摺り性悪化によるシワの発生

The property of the biaxial orientation polypropylene film of the above-mentioned examples 1-3 and a metalization biaxial orientation polypropylene film and the property of a capacitor element were shown in the above-mentioned table 1. Also when it was satisfactory and a metalization polypropylene film was processed into a capacitor element, destruction of the component after electric charging of 100 hours after was not accepted, either, but tandelta of each of vacuum evaporatio workability of a biaxial orientation polypropylene film and film plasticities was also small.

[0073] (Example 1 of a comparison) The biaxial orientation polypropylene film, the metalization biaxial orientation polypropylene film, and the capacitor element were obtained by the same approach as an example 1 except ash content having made casting drum temperature 85 degrees C using the polypropylene raw material which II considered 97.8% and mmmm considered as the same anti-oxidant formula as an example 1 by 19 ppm 98%. Similarly this property was shown in Table 1. This invention of II and mmmm is out of range, Siwa by heat defeat occurred at the time of vacuum evaporatio processing, and, 100 hours after, destruction had generated the capacitor element. The property was shown in Table 1.

[0074] (Examples 2 and 3 of a comparison) Corona discharge treatment was not performed to the biaxial orientation polypropylene film which produced the film like the example 1, but it is what made wetting tension 32 mN/m, and the metalization biaxial orientation polypropylene film and capacitor element of 6ohms / ** (example 2 of a comparison), and 22ohms / ** (example 3 of a comparison) were obtained by the vacuum plating of aluminium. Optical density was 2.6 and 0.9, respectively. Similarly the property was shown in Table 1. The biaxial orientation polypropylene film of the example 2 of a comparison had the bad film plasticity, and it became that in which the self heel nature of a metalization biaxial orientation polypropylene film was also inferior. Moreover, a capacitor element did not wait for 100 hours, either, but broke. The film

plasticity was bad, since the surface-electrical-resistance value of metalization biaxial orientation polypropylene was large, R-OD exceeded the range of this invention, tandelta at the time of considering as a capacitor element also became large, and the biaxial orientation polypropylene film of the example 3 of a comparison also destroyed it after that in 125 hours. [0075] (Example 4 of a comparison) Although it is what performed corona discharge treatment of 60 W-min/m² to the biaxial orientation polypropylene film which produced the film like the example 1, and made wetting tension 52 mN/m and was going to obtain the metalization biaxial orientation polypropylene film of 6ohms / ** by the vacuum plating of aluminium, since slipping nature is bad, Siwa occurred and blocking occurred, vacuum evaporatio no was interrupted, and processing to a capacitor element was postponed.

[0076]

[Effect of the Invention] The biaxial orientation polypropylene film by this invention should be excellent in the plasticity of the metal membrane by vacuum evaporatio no, and should be excellent in vacuum evaporatio no workability without concern of generating of Siwa. The metalization biaxial orientation polypropylene film of this invention is still more useful in order to create the capacitor element which was excellent in self heel nature and was excellent in the dielectric-breakdown-proof property over a long period of time [in an elevated temperature] when the dielectric loss at the time of considering as a capacitor element is small. According to this invention, the maximum service temperature of a polypropylene film capacitor can be raised by a maximum of 20 degrees C rather than conventional 85 degrees C by this, and, thereby, it can respond to the miniaturization of an electric apparatus, and high density-ization of a component.

[Translation done.]

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(54) 【発明の名称】 二軸配向ポリプロピレンフィルム、金属化二軸配向ポリプロピレンフィルムおよびコンデンサー

(57) 【要約】

【課題】耐熱性と蒸着加工性に優れた二軸配向ポリプロピレンフィルムと、セルフヒール性に優れた表面抵抗値の小さい金属化二軸配向ポリプロピレンフィルム、および高温での長期耐熱性を向上させたコンデンサーが提供される。

【解決手段】アイソタクチシティが 98～99.5%、アイソタクチックペンタッド分率が 99%を越え、少なくとも一方の面のぬれ張力が 35～50 mN/mである二軸配向ポリプロピレンフィルムおよびアイソタクチシティが 98～99.5%、アイソタクチックペンタッド分率が 99%を越えた二軸配向ポリプロピレンフィルムの少なくとも片面に、表面抵抗 R と光学濃度 OD の関係が $R \cdot OD < 15$ である金属層が形成された金属化フィルムおよびそれを用いたコンデンサー。

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【特許請求の範囲】

【請求項1】 二軸配向されたポリプロピレンフィルムであって、該ポリプロピレンフィルムのアイソタクチシティが98～99.5%であり、アイソタクチックペンタッド分率が99%を越え、少なくとも一方の面のぬれ張力が35～50mN/mであることを特徴とする二軸配向ポリプロピレンフィルム。

【請求項2】 アイソタクチシティが98～99.5%であり、アイソタクチックペンタッド分率が99%を越えた二軸配向ポリプロピレンフィルムの少なくとも一方の面に金属層が形成され、該金属層の表面抵抗R(Ω/□)と光学濃度ODの関係が、 $R \cdot OD < 15$

であることを特徴とする金属化二軸配向ポリプロピレンフィルム。

【請求項3】 請求項2に記載の金属化二軸配向ポリプロピレンフィルムを巻回あるいは積層してなることを特徴とするコンデンサー。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は、耐熱性に優れた二軸配向ポリプロピレンフィルム、および特定の表面抵抗と光学濃度の関係を有した金属化二軸配向ポリプロピレンフィルムおよびそれを用いたコンデンサーに関し、特に蒸着加工性に優れた二軸配向ポリプロピレンフィルム、セルフヒール性に優れた表面抵抗値の小さい金属化二軸配向ポリプロピレンフィルムおよびそれを誘電体として用いた耐熱性、耐絶縁破壊特性に優れたコンデンサーに関するものである。

【0002】

【従来の技術】二軸配向ポリプロピレンフィルムは、透明性、光沢性などの光学的特性に優れ、さらに水蒸気バリア性能や優れた電気特性などにより、包装用途、コンデンサー用途などに広範に用いられている。

【0003】この二軸配向ポリプロピレンフィルムは、フィルムコンデンサーの誘電体として用いられる代表的な素材の一つであるが、もう一つの代表的素材であるポリエステルフィルムと比較して耐熱性が低いため、コンデンサーとしての最高使用温度が85℃程度に制限されていた。その理由は、使用温度が高温になると、フィルムの非晶部、異物の影響等から、本来ポリプロピレンフィルムの特長であるべき絶縁破壊強度が急激に低下してしまい、特に長期間の使用に耐えられなくなる場合があったからである。

【0004】一方、電気装置の小型化に伴い、素子の密集化および高温化が進展し、従来のポリプロピレンフィルムコンデンサーの最高使用温度をさらに上昇させたいという要求が強くなってきている。このためには、従来のポリプロピレンフィルムコンデンサーの最高使用温度である85℃よりも高温でしかも長期に性能を維持する

必要があった。

【0005】またポリプロピレンフィルムコンデンサーは、一般にフィルム的一方の面に蒸着によりアルミや亜鉛あるいはこれらの合金からなる電極としての金属層が形成され(以下、金属化という)、この金属化フィルムを巻回あるいは積層してコンデンサー素子とすることが行われている。

【0006】従って上記工程を経た耐熱性に優れたコンデンサーを得るためには(1)高温でのフィルム電気特性が優れること、(2)蒸着による金属層が均一に形成され、セルフヒール性に優れた表面抵抗値が小さいことおよび(3)蒸着加工およびコンデンサー素子作成時の滑り性など耐加工性に優れることが求められていた。

【0007】このような課題に対し、特開平6-236709号公報には灰分が低く、沸騰n-ヘプタン可溶分が1～10重量%であることから加工性に優れ、室温から80℃までの電気絶縁性に優れた高分子絶縁材料が開示されており、沸騰n-ヘプタン不溶部のアイソタクチックペンタッド分率が90%以上のものが好ましいとの示唆がある。

【0008】また、特開平7-25946号公報には同じく沸騰n-ヘプタン不溶分が80重量%以上、特に好ましくは96重量%以上であり、該沸騰n-ヘプタン不溶成分のアイソタクチックペンタッド分率が0.970～0.995の範囲にあるプロピレン重合体およびこれを用いた成形体の開示がある。

【0009】しかし、これらに開示されたように、単に沸騰n-ヘプタン不溶分のアイソタクチックペンタッド分率の高い二軸配向ポリプロピレンフィルムでは、本発明の目指す85℃を越える高温での耐絶縁破壊特性とこのフィルムを誘電体として用いたコンデンサー素子の長期耐熱性が不十分であった。すなわち、上記の従来の技術による立体規則性の高い二軸配向ポリプロピレンフィルムは、沸騰n-ヘプタン不溶部のアイソタクチックペンタッド分率がそこそこ高いものの、n-ヘプタン可溶分のアイソタクチックペンタッド分率が低いため、フィルムとしてのアイソタクチックペンタッド分率が結果として低く、立体規則性が不十分であった。またアイソタクチシティが極めて高い、いわゆる高結晶性の二軸配向ポリプロピレンフィルムは、立体規則性が不十分であるが故に製膜性が極めて悪く、耐熱性と耐絶縁破壊特性に優れた二軸配向ポリプロピレンフィルムを製造するための工業的に有用な技術として確立されるには至っていなかった。

【0010】これらの欠点を解消するため、特公平4-28727号公報にはアイソタクチックペンタッド分率が0.960～0.990の範囲にあり、かつ沸騰n-ヘキサンおよび沸騰n-ヘプタンで逐次抽出した被抽出物の全量が3.0～6.0%とすることで成形性に優れた結晶性ポリプロピレンフィルムが提案されている。し

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かし、このポリプロピレンフィルムはアイソタクチックペンタッド分率が十分ではなく、高温での耐絶縁破壊特性が不十分であった。

【0011】さらに特開平5-217799号公報には、特定の熱変形温度とヤング率を有し、結晶化度が高く、立体規則性の良い高剛性ポリプロピレンフィルムに金属を蒸着した高剛性蒸着金属化フィルムを用いた蒸着フィルムコンデンサーが提案されている。しかし、ここでの立体規則性は高々90%程度であり、高温での絶縁破壊特性が不十分であった。

【0012】さらに特開平7-50224号公報には、120℃における熱収縮率が長さ方向で4.0%以下、幅方向で0.8%以下である金属化ポリプロピレンフィルムが開示されている。しかし、このフィルムのアイソタクチシティおよび立体規則性は従来のレベルのものであり、今後の高度な要求に対応するための、本発明の目的である高温での耐絶縁破壊特性が必ずしも十分とは言えなかった。

【0013】

【発明が解決しようとする課題】本発明者等は、ポリプロピレンフィルムのアイソタクチシティと立体規則性を高度に制御し、表面のぬれ張力を適正化し、金属化層の表面抵抗と光学濃度を制御することで、従来技術では達成し得なかった耐熱性、耐絶縁破壊特性に優れたコンデンサーを得ることができることを見出し、本発明に至った。

【0014】本発明の目的は、耐熱性に優れ、蒸着加工性に優れた二軸配向ポリプロピレンフィルムを提供することにある。

【0015】本発明の他の目的は、セルフヒール性に優れた表面抵抗値の小さい金属化二軸配向ポリプロピレンフィルムおよびそれを用いた耐熱性、耐絶縁破壊特性に優れたコンデンサーを提供することにある。

【0016】

【課題を解決するための手段】上記目的を達成する本発明のポリプロピレンフィルムは、二軸配向されたポリプロピレンフィルムであって、そのポリプロピレンフィルムのアイソタクチシティが98~99.5%であり、アイソタクチックペンタッド分率が99%を越え、少なくとも一方の面のぬれ張力が35~50mN/mであることを特徴とするものである。

【0017】また、本発明の金属化二軸配向ポリプロピレンフィルムは、アイソタクチシティが98~99.5%であり、アイソタクチックペンタッド分率が99%を越えた二軸配向ポリプロピレンフィルムの少なくとも一方の面に金属層が形成され、その金属層の表面抵抗R(Ω/□)と光学濃度ODの関係が、 $R \cdot OD < 15$ であることを特徴とするものであり、または、アイソタクチシティが98~99.5%であり、アイソタクチックペンタッド分率が99%を越え、少なくとも一方の面の

ぬれ張力が35~50mN/mである二軸配向ポリプロピレンフィルムの少なくとも一方の面に、表面抵抗R(Ω/□)と光学濃度ODの関係が、 $R \cdot OD < 15$ である金属層が形成されてなることを特徴とするものである。

【0018】さらに本発明のコンデンサーは、かかる金属化二軸配向ポリプロピレンフィルムを巻回あるいは積層してなるものである。

【0019】

10 【発明の実施の形態】本発明の二軸配向ポリプロピレンフィルムは、主としてポリプロピレンの単独重合体からなる二軸配向フィルムであるが、本発明の目的を阻害しない範囲で、他の不飽和炭化水素による共重合成分などを含有していてもよく、また他の重合体がブレンドされていてもよい。共重合成分としては、例えばエチレン、1-ブテン、1-ペンテン、3-メチルペンテン-1、3-メチルブテン-1、1-ヘキセン、4-メチルペンテン-1、5-エチルヘキセン-1、1-オクテン、1-デセン、1-ドデセン、ビニルシクロヘキセン、スチレン、アリルベンゼン、シクロペンテン、ノルボルネン、5-メチル-2-ノルボルネンなどが挙げられる。共重合量は、蒸着加工性、耐絶縁破壊特性、耐熱性の点から1mol%未満、ブレンド物は1wt%未満が好ましい。

【0020】本発明において、二軸配向ポリプロピレンフィルムのアイソタクチシティは、特に二軸延伸時の幅方向延伸性の点で99.5%以下である。ここでアイソタクチシティとは、フィルムを沸騰n-ヘプタンで抽出した場合の、抽出前フィルム重量に対する不溶分の重量の割合により定義される。アイソタクチシティが高すぎると、特開平6-236709号公報にあるように二軸延伸フィルムを製造する際、延伸性が悪く、製膜が著しく困難となる。また特に蒸着加工性、さらに詳しくはフィルム厚みが5μm未満となった場合の蒸着時の耐熱負け性の点でアイソタクチシティは98%以上である。このようにアイソタクチシティが98%以上、99.5%以下であれば、フィルム厚みが5μm未満のように非常に薄いフィルムの場合でも良好な延伸ができ、かつ、蒸着時の耐熱負け性において高レベルを維持することができ、薄いフィルムを使つての小型化を可能とし、従来にはない高性能なコンデンサーを実現できるのである。アイソタクチシティが小さすぎると耐熱性に劣り、蒸着時の熱負けによるシワが発生する場合がある。良好な製膜性と蒸着加工性、耐絶縁破壊特性のためにより好ましいアイソタクチシティは98.7~99.5%であり、さらには98.7~99.3%が好ましい。

【0021】このようなアイソタクチシティを有する二軸配向ポリプロピレンフィルムとするには、原料であるポリプロピレン樹脂の沸騰n-ヘプタンに溶けやすい低分子量成分や、立体規則性の低い、いわゆるアタクチック

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クの部分の割合が適度に低いものをのを選択するなどの方法を採用することができる。

【0022】本発明において、二軸配向ポリプロピレンフィルムの立体規則性は、 ^{13}C -NMRにより測定したメチル基の吸収ピークによるペンタッド分率により評価することができる。一般的に、ポリプロピレン分子鎖における5個の繰り返し単位(ペンタッド)の立体配座には、mmmm、mmmr、rmmr、 \cdots 、rrrr、 \cdots 、mrrr、mrrm等がある。ここで、mはメソ(meso)、rはラセモ(rasemo)の立体配座を示す。

【0023】二軸配向ポリプロピレンフィルムのペンタッド分率は、例えばT. Hayashiらの報告[Polymer, 29, 138~143 (1988)]等にあるように、上記各立体配座を有するセグメントの比率を ^{13}C -NMRから求めることができる。これらのうち、全メチル基の吸収強度に対するmmmmの立体配座の割合、すなわちアイソタクチックペンタッド分率(以下mmmmと省略する場合がある)は、m(mmmm)m、m(mmmm)r、r(mmmm)rの3つのヘブタッド分率の和として定義される。本発明の二軸配向ポリプロピレンフィルムのアイソタクチックペンタッド分率mmmmは、99%を越える。このようなフィルムは、極めて長いアイソタクチックセグメントを持つ分子から構成されたポリプロピレンからなっているため、蒸着可能性に優れた、高結晶性、高耐熱性、高耐絶縁破壊特性のフィルムを与え得る。

【0024】本発明の二軸配向ポリプロピレンフィルムのmmmmは、蒸着加工性、高耐熱性、高耐絶縁破壊特性の点で好ましくは99.1%以上であり、より好ましくは99.2%以上であり、さらに好ましくは99.3%以上である。

【0025】このような立体規則性を付与するには、原料であるポリプロピレン樹脂の立体規則性を高度に制御することが有効である。このような原料を作成する方法としては、ポリプロピレンを重合する際の、触媒系(固体触媒、外部添加電子供与性化合物)やこれらの純度により達成される。原料のポリプロピレン樹脂のmmmmが高いものほど二軸配向ポリプロピレンフィルムのmmmmが高くなる傾向が認められるが、原料の押出系内での極度の熱劣化もmmmmを低下させるため、高温押出系での原料の長時間滞留を避けるなどの構造的工夫、押出条件が適宜選択される。

【0026】また、本発明の二軸配向ポリプロピレンフィルムにおいて、その少なくとも一方の表面のぬれ張力は35~50 mN/mである。ぬれ張力が小さすぎると電極として用いる金属層が均一に形成され難く、電極部分の電気抵抗が大きくなり、コンデンサー素子に加工した際、誘電損失が大きくなり、発熱によりさらに劣化が加速されコンデンサー素子としての寿命が短くなる場合

がある。一方、ぬれ張力が大きすぎるとフィルムの滑り性が悪くなり、蒸着加工時シワが入るなどの欠陥になり、素子作成時不都合を生じ、素子の特性を悪化させる場合がある。ぬれ張力のさらに好ましい範囲は37~48 mN/mであり、最も好ましくは38~45 mN/mである。

【0027】本発明において、表面のぬれ張力をこのような範囲とするためには、コロナ放電処理やプラズマ処理が好ましく適用でき、大気雰囲気中、窒素雰囲気中、炭酸ガス雰囲気中やこれらの混合雰囲気中でのコロナ放電処理が生産性の観点から適当であり、製膜工程、蒸着の前工程いずれで実施してもよい。処理強度は5~50 W \cdot min/m²の範囲で採用されるが、処理雰囲気により処理効果が異なるため、本発明のぬれ張力とするために、適宜処理強度を調整することが必要である。

【0028】本発明の実施態様において、二軸配向ポリプロピレンフィルムに金属層を形成して金属化二軸配向ポリプロピレンフィルムに加工することは、コンデンサー素子の小型化のために好ましく採用される。この場合、金属層を構成する金属は特に限定されることはないが、アルミニウム、亜鉛、銅、錫、銀、ニッケル等を単独または併用で使用することが金属化層の耐久性、生産性の点で好ましく、経済性の点から特にアルミニウムや亜鉛などが好ましく用いられる。

【0029】本発明の二軸配向ポリプロピレンフィルムに金属層を形成する方法は、真空蒸着法、スパッタリング法およびイオンビーム法等が挙げられるが、特に限定されない。

【0030】本発明において、金属化二軸配向ポリプロピレンフィルムの好ましい表面抵抗値Rは、1~20 Ω/\square の範囲であり、より好ましくは1.2~15 Ω/\square である。膜抵抗値が小さすぎると、蒸着膜の厚みが厚く蒸着時に熱負けが生じアバタ状の表面欠点や4 μm 前後の薄いフィルムでは穴アキ等が発生することがある。また、膜抵抗値が大きすぎると表面抵抗値が大きくなり、コンデンサー素子としての誘電損失が大きくなり、素子の破壊に繋がり易い。膜抵抗値をこの範囲とするには、蒸着時の膜抵抗値のモニターにより制御する方法が好ましく採用される。

【0031】本発明の二軸配向ポリプロピレンフィルムの光学濃度ODは、0.5~3の範囲で選択されることが好ましい。さらに好ましくは光学濃度は0.8~2の範囲である。本発明において光学濃度とは、可視域の特定波長における光線透過率の逆数の対数で定義される。光学濃度が大きすぎると蒸着時の熱負けの問題が生じることがある。光学濃度が小さすぎると表面抵抗値が大きくなる問題が生じる場合がある。

【0032】本発明においては、表面抵抗値Rと光学濃度ODとが、 $R \cdot OD < 15 (\Omega/\square)$ の関係にあることが重要である。ODは蒸着金属の膜厚に比例して大き

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【００４１】ポリプロピレンの重合過程においては、金属を含む化合物を触媒として用い、必要に応じ重合後にこの残礎を除去することが一般的であるが、この残礎は樹脂を完全に燃焼させた残りの金属酸化物の量を求めることで評価でき、これを灰分と呼ぶ。本発明の二軸配向ポリプロピレンフィルムの灰分は３０ｐｐｍ以下であることが好ましく、より好ましくは２５ｐｐｍ以下であり、さらに好ましくは２０ｐｐｍ以下である。灰分が多すぎると、フィルムの耐熱縁破壊特性が低下し、これを

用いたコンデンサーの絶縁破壊強度が低下する場合がある。灰分をこの範囲にするには、触媒残燼の少ない原料を用いることが重要であるが、製膜時の押出系からの汚染も極力低減するなどの方法、例えばブリード時間を1時間以上かけるなどの方法を採用することができる。

【0042】本発明の二軸配向ポリプロピレンフィルムおよび金属化二軸配向ポリプロピレンフィルムの表面粗さは目的に応じて適宜選択されるが、中心線平均粗さで0.02~0.2μmの範囲が好ましい。中心線平均粗さが大きすぎると、フィルムを積層した場合に層間に空気が入りコンデンサー素子の劣化に繋がる場合がある。逆に小さすぎるとフィルムの滑りが悪くなり、ハンドリング性に劣る場合がある。

【0043】本発明の二軸配向ポリプロピレンフィルムおよび金属化二軸配向ポリプロピレンフィルムには、公知の添加剤、例えば結晶核剤、酸化防止剤、熱安定剤、すべり剤、帯電防止剤、ブロッキング防止剤、充填剤、粘度調整剤、着色防止剤などを含有させてもよい。

【0044】これらの中で、酸化防止剤の種類および添加量の選定は、長期耐熱性にとって重要である。本発明の二軸配向ポリプロピレンフィルムおよび金属化二軸配向ポリプロピレンフィルムに添加される酸化防止剤は立体障害性を有するフェノール性のもので、そのうち少なくとも1種は分子量500以上の高分子量型のものが溶解時の飛散防止のために好ましい。この具体例としては種々のものが挙げられるが、例えば2,6-ジ-tert-ブチル-p-クレゾール(BHT:分子量220.4)とともに1,3,5-トリメチル-2,4,6-トリリス(3,5-ジ-tert-ブチル-4-ヒドロキシベンジル)ベンゼン(例えばチバガイギー社製Irganox1330:分子量775.2)またはテトラキス[メチレン-3-(3,5-ジ-tert-ブチル-4-ヒドロキシフェニル)プロピオネート]メタン(例えばチバガイギー社製Irganox1010:分子量1177.7)等を併用することが好ましい。これら酸化防止剤の総含有量は、ポリプロピレン全量に対して0.03~1重量%(300~1000ppm)の範囲が好ましい。総含有量が少ないと長期耐熱性に劣る場合があり、多すぎるとこれら酸化防止剤のブリードアウトによる高温下でのブロッキングにより、コンデンサー素子に悪影響を及ぼす場合がある。より好ましい含有量は0.1~0.9重量%であり、さらに好ましくは0.2~0.8重量%である。

【0045】本発明の金属化二軸配向ポリプロピレンフィルムのセルフヒール性は、直流電圧を印加した場合の、部分的な放電破壊の様子で評価できる。部分的な放電破壊後の蒸着金属膜の蒸発領域が小さく、放電痕の周辺に蒸着金属が残存しない場合セルフヒール性は良好である。放電による放電痕の周辺に金属が残存し、フィルムが溶解した様子が観察されるものはセルフヒール性が悪く、コンデンサーに加工した場合、素子そのものの絶

縁破壊に至る場合がある。特に105℃での課電時には絶縁破壊が加速される場合がある。

【0046】本発明のコンデンサーの特性は、105℃で100時間の定格電圧の1.2~1.3倍の交流電圧課電後の誘電損失大きさ、すなわちtanδで評価できる。tanδが大きすぎると素子の発熱が加速され短時間で破壊に至る場合がある。本発明の二軸配向ポリプロピレンフィルム、金属化二軸配向ポリプロピレンフィルムおよびそれからなるコンデンサーの製造方法を以下に説明するが、本発明は必ずしもこれに限定されるものではない。

【0047】まず、ポリプロピレン原料を押出機に供給し、加熱熔融し、濾過フィルターを通させた後、220~320℃の温度でスリット状口金から熔融押し出し、40~85℃の温度に保たれたキャストドラムに巻き付けて冷却固化せしめ、未延伸フィルムを作る。

【0048】次に、この未延伸フィルムを二軸延伸して二軸配向せしめる。延伸方法としては逐次二軸延伸方法が好ましい。逐次延伸方法は、まず未延伸フィルムを120~150℃に保たれたロールに通して予熱し、引き続きそのシートを140℃~150℃の温度に保ち周速差を設けたロール間に通し、長手方向に2~6倍に延伸し、ただちに室温に冷却する。ここで、本発明のmmmmが99%を超える二軸配向ポリプロピレンフィルムは、予熱温度130℃以下、延伸温度140℃以下では熱量が不足して延伸ムラを起こしたり破けて製膜できない場合があり、140℃を超える延伸温度を採用することが重要である。引き続きその延伸フィルムをテンターに導いて、160~165℃の温度で幅方向に5~10倍に延伸し、次いで幅方向に2~20%の弛緩を与えつつ、150~160℃の温度で熱固定して巻取る。本発明において、この熱固定の温度は重要であり熱固定温度が低すぎると熱収縮率が大きくなり、本発明の範囲を超える場合がある。

【0049】その後、蒸着を施す面に蒸着金属の接着性を良好ならしめるために、空气中、窒素中、炭酸ガス中あるいはこれらの混合気体中でコロナ放電処理を行ないワインダーで巻取る。

【0050】次に得られたフィルムを真空蒸着装置にセットし、目的に応じた金属を、所定の膜抵抗に蒸着する。この蒸着フィルムをスリットし、コンデンサー素子を作るための2リール一対の蒸着リールとする。この後、素子状に巻回し、熱プレスして扁平状に成形し、端部の金属溶射、リード取り出し、外装を経てコンデンサーとする。

【0051】本発明の二軸配向ポリプロピレンフィルムは、上記コンデンサー用途以外に、低い熱収縮率を生かして、蒸着、印刷、ラミネート、ヒートシールなどの加工時の熱による変形を抑えることができるため、種々の包装用途として、例えばこれにヒートシール層とラミネ

ートして使用でき、また粘着テープやつや出しフィルム（プリントラミネート）などとしても好適に用いることができる。

【００５２】本発明における特性値の測定方法、並びに評価方法は次のとおりである。

【0053】(1) アイソタクチシティ (アイソタクチックインデックス: 11)

試料を60℃以下の温度のn-ヘプタンで2時間抽出し、ポリプロピレンへの添加物を除去する。その後130℃で2時間真空乾燥する。これから重量W (mg) の試料を取り、ソックスレー抽出器に入れ沸騰n-ヘプタンで12時間抽出する。次に、この試料を取り出しアセトンで十分洗浄した後、130℃で6時間真空乾燥しその後常温まで冷却し、重量W' (mg) を測定し、次式で求めた。

$$II = (W' / W) \times 100 (\%)$$

(2) アイソタクチックペンタッド分率

試料を o -ジクロロベンゼンに溶解し、J EOL製JNM-GX270装置を用い、共鳴周波数67.93MHzで ^{13}C -NMRを測定した。得られたスペクトルの帰属およびベンタッド分率の計算については、T. Hayashiらが行った方法[*Polymer*, 29, 138~143 (1988)]に基づき、メチル基由来のスペクトルについて、mmmmmmピークを21.855 ppmとして各ピークの帰属を行ない、ピーク面積を求めてメチル基由来全ピーク面積に対する比率を百分率で表示した。詳細な測定条件は以下のとおりである。

【0054】測定溶媒：o-ジクロロベンゼン(90wt%)
/ベンゼン-D6(10wt%)

測定温度 : 120~130℃

共鳴周波数：67.93MHz

パルス幅 : 10 μ sec (45° パルス)

パルス繰り返し時間：7.091 sec

データ点 : 32K

積算回数 : 8 1 6 8

測定モード：ノイズデカップリング

(3) フィルム厚み

ダイヤルゲージ式厚み計（JIS-B-7503）を用いて測定した。

【0055】(4)ぬれ張力

JIS K-6788に従って測定した。

【0056】(5) 金属膜形成性

二軸配向ポリプロピレンフィルムの蒸着加工後の蒸着膜の膜形成性を目視で評価し以下の判定を行なった。

【0057】○：金属膜にむらがなく金属膜が均一に形成されている。

〔0058〕×：金属膜にむらがある。

(0059) (6) 蒸着加工性

二軸配向ポリプロピレンフィルムの蒸着加工後の巻上がりのロールの状態を目視で評価し以下の判定を行なっ

た。

【0060】○：ロールにシワが発生していない。

【0061】×：ロールにシワが発生している。

【0062】(7) 光学濃度OD

光学濃度計（マクベス社製TR 9 2 7）を用い、フィルターをVisualとしたときの透過濃度を測定し、金属化フィルムの光学濃度から原反フィルムの光学濃度を差し引いた値を金属層の光学濃度とした。

【0063】(8) 表面抵抗R

10 表面抵抗計（三菱油化 Loresta-FP）を用い、4端子プローブを用いて測定した。

【0064】(9)セルフヒール性

金属化二軸配向ポリプロピレンフィルムの金属化を行っていない面を平坦な黄銅製の電極板に接触させ、金属化面と黄銅製電極板の間に定格電圧（ポリプロピレンフィルムの場合は $4.5 \sim 5.0 \text{ V}/\mu\text{m}$ ）の倍の直流電圧を印加し部分的な絶縁破壊を発生させた。絶縁破壊箇所の観察を行ない、以下のセルフヒール性の判定を行った。

20 【0065】○：絶縁破壊によりフィルムに形成された孔の直径が1mm以下で孔の周辺に金属が認められない。

【0066】×：孔の直径が3 mm以上でフィルムの溶解の痕が認められる。

【0067】△：上記の中間

(10) $\tan \delta$ 誘電損失

105℃に保持されたコンデンサー素子を、春日電気（株）製交流高圧安定化電源に接続し、60V/μmの電圧を印加し100時間後のtanδをシェーリングブリッジ法で電圧250Vで測定した。

(0068)

【実施例】本発明を実施例、比較例に基づいて以下に詳細に説明する。

【0069】（実施例1）アイソタクチックインデックス11が98.8%、アイソタクチックペンタッド分率mmmmが99.5%、灰分が19ppmのポリプロピレン原料に2,6-ジ-*t*-ブチル-*p*-クレゾール（BHT）0.3%、テロラキス[メチレン-3（3,5-ジ-*t*-ブチル-4-ヒドロキシフェニル）プロピオネート]メタン（Irganox1010）0.5%を添加したものを押出機に供給して280℃の温度で熔融し、T型口金からシート状に押出成形し、70℃の温度のキャスティングドラムに巻き付けて冷却固化した。次いで、このシートを135℃で予熱し、引き続き143℃の温度に保ち周速差を設けたロール間に通し、長手方向に5倍に延伸した。引き続きそのフィルムをテンターに導き、162℃の温度で幅方向に10倍延伸し、次いで幅方向に8%の弛緩を与えながら158℃で熱処理を行ない4μmの厚みの二軸配向ポリプロピレンフィルムを得た。さらに30W・min/m²の処理強度で大気中でコロ

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0 た、

【表 1】

【表 1】

	II (%)	mm (%)	ぬれ張力 (mN/m)	蒸着加工性	膜形成性	表面抵抗 (Ω/□)	光学透過度	R・OD (Ω/□)	熱安定性	tan δ
実施例1	98.8	99.5	41	○	○	6	1.8	10.8	○	0.02
実施例2	"	"	38	○	○	6	2.0	12	○	0.02
実施例3	"	"	41	○	○	11	1.2	13.2	○	0.04
比較例1	98.3	98	41	×	○	6	1.9	11.4	○	破壊
比較例2	99.8	99.5	32	○	×	6	2.6	15.6	×	破壊
比較例3	"	"	"	○	×	2.2	0.9	19.8	○	0.15
比較例4	"	"	52	× [△]	—	—	—	—	—	—

＊ ＊ 滑り性悪化によるシワの発生

＊ ＊ 滑り性悪化によるシワの発生

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【００７１】（実施例３）実施例１の４１ｍＮ／ｍのぬれ張力の二軸配向ポリプロピレンフィルムを用いて、アルミニウムを蒸着し、光学濃度１．２の金属化フィルムを得た。また、表面抵抗は１１Ω／□であった。これを用いて、実施例１と同様にコンデンサー素子を作成し

た。

(0072)

【表 1】

ヲム蒸着で $6\Omega/\square$ (比較例 2) と $22\Omega/\square$ (比較例 3) の金属化二軸配向ポリプロピレンフィルムとコンデンサー素子を得た。光学濃度はそれぞれ 2.6 と 0.9 であった。同じく特性を表 1 に示した。比較例 2 の二軸配向ポリプロピレンフィルムは膜形成性が悪く、金属化二軸配向ポリプロピレンフィルムのセルフヒール性も劣ったものとなった。またコンデンサー素子も 100 時間を待たず破壊した。比較例 3 の二軸配向ポリプロピレンフィルムも膜形成性が悪く、金属化二軸配向ポリプロピレンの表面抵抗値が大きいために R・OD が本発明の範囲を超え、コンデンサー素子とした場合の $\tan \delta$ も大きくなり、その後 125 時間で破壊した。

【００７５】（比較例４）実施例１と同様に製膜した二軸配向ポリプロピレンフィルムに $60\text{ W} \cdot \text{min} / \text{m}^2$ のコロナ放電処理を施し、ぬれ張力を $52\text{ mN} / \text{m}$ としたものでアルミニウム蒸着で $6\ \Omega / \square$ の金属化二軸配向ポリプロピレンフィルムを得ようとしたが、滑り性が悪くシワが発生し、ブロッキングが発生したために蒸着を中断し、コンデンサー素子への加工は見合わせた。

(0 0 7 6)

(9)

特開平10-119126

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【発明の効果】本発明による二軸配向ポリプロピレンフィルムは、蒸着による金属膜の形成性に優れ、シワの発生懸念のない蒸着加工性に優れたものとしてすることができる。さらに本発明の金属化二軸配向ポリプロピレンフィルムは、セルフヒール性に優れ、コンデンサー素子とした場合の誘電損失の小さい、高温での長期耐絶縁破壊*

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* 特性に優れたコンデンサー素子を作成するために有用である。このことにより本発明によれば、ポリプロピレンフィルムコンデンサーの最高使用温度を従来の85℃よりも最高20℃向上させることができ、これにより電気装置の小型化、素子の密集化に対応することができる。

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